INVITED REVIEW

Magnetic polymer nanocomposites for environmental and biomedical applications

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Abstract Hybrid nanomaterials have received voluminous interest due to the combination of unique properties of organic and inorganic component in one material. In this class, magnetic polymer nanocomposites are of particular interest because of the combination of excellent magnetic properties, stability, and good biocompatibility. Organic–inorganic magnetic nanocomposites can be prepared by in situ, ex situ, microwave reflux, co-precipitation, melt blending, and ceramic–glass processing and plasma polymerization techniques. These nanocomposites have been exploited for in vivo imaging, as superparamagnetic or negative contrast agents, drug carriers, heavy metal adsorbents, and magnetically recoverable photocatalysts for degradation of organic pollutants. This review article is mainly focused on fabrication of magnetic polymer nanocomposites and their applications. Different types of magnetic nanoparticles, methods of their synthesis, properties, and applications have also been reviewed briefly. The review also provides detailed insight into various types of magnetic nanocomposites and their synthesis. Diverse applications of magnetic nanocomposites including environmental and biomedical uses have been discussed.

Introduction

During the last decade, the development of magnetic nanocomposite materials has been the source of discovery of spectacular new phenomena, with potential applications in the multidimensional fields. Among the broad spectrum of nanoscale materials being investigated for various environmental and biomedical applications, magnetic nanoparticles (MNPs) have gained significant attention due to their intrinsic magnetic properties, which makes them successful as magnetically recoverable catalysts, drug delivery agents, anticancer materials, magnetic resonance imaging devices, etc. This class of nanomaterials includes metallic, bimetallic nanoparticles, metal oxides, ferrites, and superparamagnetic iron oxide nanoparticles (SPIONs) [1, 2]. Magnetic nanoparticles and nanocomposites have aroused significant scientific and technological interest because of their potential applications in the fields of biomedicine, information technology, magnetic resonance imaging, catalysis, telecommunication, and environmental remediation [3–6]. Magnetic nanocomposites generally comprise of magnetic nanoparticles embedded in nonmagnetic or magnetic matrix [7, 8]. However, magnetic nanoparticles dispersed in composites usually have a strong tendency to form agglomerates for reduction of energy associated with high surface area-to-volume ratio of the nanosized particles. To avoid aggregation of magnetic nanoparticles, protection strategies have been developed to chemically stabilize the

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naked magnetic nanoparticles by grafting of or coating with organic species, including surfactants or polymers, or coating with an inorganic layer, such as silica or carbon. The incorporation of these functionalized magnetic nanoparticles in polymer or other matrices for the development of magnetic nanocomposite materials proved to be more effective [9, 10]. There are mainly four types of magnetic nanocomposites, i.e., core–shell inorganic nanocomposites, self-assembled nanocomposites, silica-based magnetic nanocomposites, and organic–inorganic nanocomposites. Among these, organic–inorganic magnetic nanocomposites have gained more interest due to combination of unique properties of organic and inorganic component in one material. Hybrid organic–inorganic magnetic nanocomposite materials can be prepared by in situ, ex situ, microwave reflux, co-precipitation, melt blending, ceramic–glass processing, and plasma polymerization techniques.

This review article is focused on synthesis, properties, and applications of magnetic nanoparticles and their polymer nanocomposites. In the first part, we have briefly reviewed different types of magnetic nanoparticles and various methods for their synthesis, properties, and applications. In the second part, we have discussed about various types of magnetic nanocomposites and methods of their synthesis. In the third part, biomedical and environmental applications of magnetic nanocomposites materials have been reported in detail.

Magnetic nanoparticles

Types of magnetic nanoparticles

Metal and metal oxide nanoparticles

Transition metals such as iron (Fe), Ni, and Co are a class of magnetic materials which are studied for their wide range of applications in various fields. These magnetic metals exhibit ferromagnetism at low temperatures and room temperature and show paramagnetism at high temperature. Studies on synthesis and magnetic properties of Fe, Ni, and Co nanoparticles have been reported in literature [11–14]. Furthermore, oxides of these metals are also known for their magnetic behavior. Among these, iron oxide nanoparticles are most important because of their biomedical and industrial applications. Iron oxides are compounds which are composed of Fe together with O and or OH. There are 16 iron oxides which are oxides, hydroxides, or oxide-hydroxides. The three main types of iron oxides are hematite, magnetite, and maghemite; out of which, magnetite has the highest saturation magnetization [15].

Ferrites

Ferrites belong to nonconductive class of ferromagnetic materials derived from metal oxides as α-Fe_2O_3, magnetite

(Fe_3O_4), etc. Ferrites have three different structural symmetries: garnet, hexagonal, and cubic or spinel ferrites which are determined by the size and charge of the metal ions that balance the charge of the oxygen ions and their relative amounts [16–21]. Many researchers have reported about the diameter and magnetic domains of ferromagnetic particles [22–24].

Among magnetic nanoparticles, SPIONs are the most exploited ones. The unique magnetic properties of SPIONs arise from a combination of their atomic composition, crystal structure, and size effects. SPIONs can produce heat by the loss mechanism which is obtained from the rotation of magnetic moments in overcoming the energy barrier. Energy is generated by the relaxation of the MNP moment to its equilibrium orientation (i.e., Neel relaxation) [25]. As per definition, hysteresis is zero for superparamagnetic materials. But in real SPION ensembles, a hysteresis loop with a negligible remanence and coercivity occurs [26] probably due to some large particles and agglomerates in the ensemble.

Dilute magnetic semiconductors

Dilute magnetic semiconductors (DMS) are a class of materials that possess the characteristics of semiconductors, as well as magnetic properties. In DMS, a fraction of the cations in the lattice are substituted by magnetic ions, and atomic spin on these magnetic dopants is expected to interact with the carriers in the lattice to bring about global ferromagnetic order in the material. Thus, these materials have unusual magnetic characteristics due to the presence of isolated magnetic ions in semiconducting lattice. Significant effort was made in trying to develop various DMS candidates as well as in understanding the origin of magnetism in these materials starting from the 1980s. The DMS includes simple oxides like SnO_2 [27–29], ZnO [30–32], TiO_2 [33, 34], or mixed oxides [35, 36] doped with several transition metals (Fe, Co, Ni, Mn) [27, 28] or rare earths metals (Dy, Eu, Er) [29].

Polymer magnets

A polymer magnet or plastic magnet is a nonmetallic magnet made from an organic polymer. This is a new class of magnetic materials which has gained the interest of researchers. Torrance et al. [37] synthesized poly(1,3,5-triaminobenzene) which when oxidized with iodine was reported to show a ferromagnetic phase up to 400 °C. After that, Rajca et al. [38] reported synthesis of organic pi-conjugated polymer with very large magnetic moment and magnetic order at low temperatures below 10 K.

In 2004, Zaidi et al. [39] reported the synthesis of a novel magnetic polymer PANiCNQ produced from polyaniline (PANi) and an acceptor molecule, tetracyanoquinodimethane (TCNQ), the first magnetic polymer to function at room

temperature. PANiCNQ combines a fully conjugated nitrogen containing backbone with molecular charge transfer side groups, and this combination gives rise to a stable polymer with a high density of localized spins which are expected to give rise to coupling. Magnetic measurements suggest that the polymer is ferri- or ferromagnetic with a curie temperature of over 350 K and a maximum saturation magnetization of 0.1 JT^{-1} kg^{-1}. Crayston et al. [40] have also reviewed the synthesis of organic magnets and other developments in the field of organic magnets.

Synthesis of magnetic nanoparticles

The synthesis of MNPs of different compositions and phases, including iron oxides, such as Fe_3O_4 and α-Fe_2O_3 [41–43], pure metals, such as Fe and Co [44, 45], spinel ferrites, such as $MgFe_2O_4$, $MnFe_2O_4$, $CoFe_2O_4$, and $NiFe_2O_4$, $ZnFe_2O_4$ [46, 47] alloys, such as $CoPt_3$ and FePt, dilute magnetic semiconductors, and polymer magnets [39, 48–50] has been reported in the literature. During the past few years, much research has been carried out to develop efficient synthetic routes for the synthesis of shape-controlled, highly stable, and monodisperse magnetic nanoparticles. A variety of methods including hydrothermal techniques, sol–gel processing, surfactant-assisted synthesis, co-precipitation, microemulsion techniques, and solution combustion have been reported in the literature for synthesis of high-quality magnetic nanoparticles.

Hydrothermal technique

Solvothermal methods have also been reported in the literature for the synthesis of MNPs and ultrafine powders [51–55]. In this technique, reactions are carried out in an aqueous media in reactors or autoclaves at high vapor pressure (generally in the range from 0.3 to 4 MPa) and high temperature (generally in the range from 130 to 250 °C). This process has also been utilized to grow dislocation free single crystal particles, and grains formed in this process could have a better crystallinity than those from other methods. Therefore, one can obtain highly crystalline magnetic nanoparticles using hydrothermal technique [51]. Further hydrothermal synthesis of magnetic nanoparticles can be achieved with or without use of specific surfactants. For example, Wang et al. [56] carried out one-step hydrothermal synthesis of highly crystalline magnetite nanoparticles (40 nm) without using surfactants, and Togashi et al. [57] reported surfactant-assisted hydrothermal synthesis of superparamagnetic magnetite nanoparticles (20 nm) at 200 °C in the presence of 3,4-dihydroxyhydroxysinnamic acid.

Recently, Phumying et al. [58] reported a novel hydrothermal method for the synthesis of spinel ferrite MFe_2O_4 (M=Ni, Co, Mn, Mg, Zn) nanocrystalline powders of different morphologies using $Fe(acac)_3$, $M(acac)_3$ (M=Ni, Co, Mn, Mg, Zn), and aloe vera plant-extracted solution. The crystal

structure and morphology of the spinel ferrite powders, as revealed by TEM, show that the $NiFe_2O_4$ and $CoFe_2O_4$ samples contain nanoparticles, whereas the $MnFe_2O_4$ and $MgFe_2O_4$ samples consist of many nanoplatelets and nanoparticles. However, $ZnFe_2O_4$ sample contains plate-like structure of networked nanocrystalline particles (Fig. 1).

Several authors have reported the hydrothermal synthesis of various magnetic nanoparticles such as iron oxide nanoparticles [51–55, 59–68], ferrites [58, 68–77], and dilute magnetic semiconductors [78].

Sol–gel method

Sol–gel is a solution chemistry-based technique to synthesize pure, stoichiometric, and monodisperse oxide nanoparticles including iron oxide nanoparticles. This technique is based on the hydrolysis of liquid precursors and formation of colloidal sols. Metal precursors, metal, or metalloid element surrounded by various reactive ligands are the starting materials that
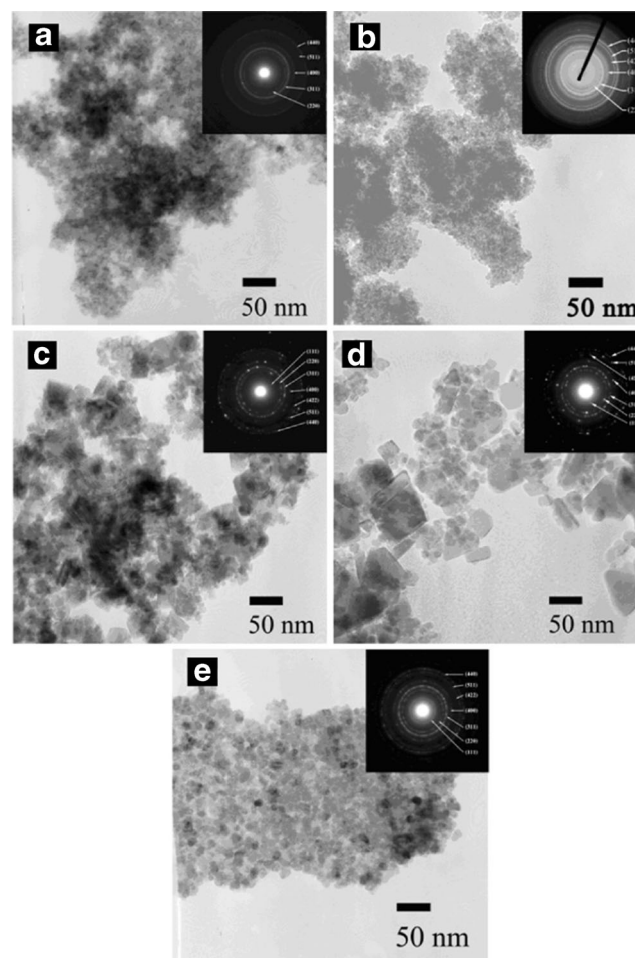
Fig. 1 TEM image with corresponding selected area electron diffraction (SAED) patterns (*inset*) of the prepared ferrite samples: **a** $NiFe_2O_4$, **b** $CoFe_2O_4$, **c** $MnFe_2O_4$, **d** $MgFe_2O_4$, and **e** $ZnFe_2O_4$. Reprinted from [58], Copyright 2013, with permission from Elsevier

undergo slow hydrolysis and polycondensation reactions to form a colloidal system named sol. The sol evolves and leads to the formation of a network containing a liquid phase, called gel [79]. This method can be performed at low temperatures and is suitable for large-scale production of nanoparticles with relatively narrow size distribution [80]. Many researchers have successfully synthesized magnetic nanoparticles by sol–gel method [81–93]. Er-doped SnO_2 and Ni-doped ZnO-DMS nanoparticles were also synthesized by sol–gel method [94, 95].

Solution combustion method

Solution combustion technique (SCT) or self-propagating high-temperature synthesis is an effective energy-saving method for the production of a variety of advanced materials including magnetic materials. This technique involves a self-sustained reaction between an oxidizer (e.g., metal nitrate) and a fuel (e.g., glycine, hydrazine). First, reactants were dissolved in water and the obtained solution thoroughly mixed, to reach essentially molecular level homogenization of the reaction medium. After being preheated to the boiling point of water and its evaporation, the solution can be ignited or it self-ignites, and the temperature rises rapidly (up to 104 °C/s) to values as high as 1,500 °C. Simultaneously, this self-sustained reaction converts the initial mixture typically to fine well-crystalline powders of desired compositions [96].

As can be seen by examining the cited articles, Patil and Sureh [97] were the first scientists to publish the instant synthesis of maghemite (γ-Fe_2O_3) by combustion process.

Deshpande et al. [96] and Erri et al. [98] applied solution combustion approach for direct synthesis of different iron oxide phases, i.e., α- and γ-Fe_2O_3 and Fe_3O_4 for the first time in literature, using a variety of fuels, viz. glycine, hydrazine, and citric acid.

After that, a number of studies on iron oxide nanomaterials and other magnetic nanoparticles synthesis by solution combustion technique have been reported in the literature [99–101]. Ahmadipour et al. [102] investigated the synthesis of nanosized $Mg_{0.2}Fe_{0.8}O$ by eco-friendly solution combustion synthesis using magnesium and iron nitrates as oxidizers and the glycine as a fuel to drive the reaction. Various stages of solution combustion process are shown in Fig. 2.

Yang et al. [103] developed a novel salt-assisted solution combustion method for the synthesis of well-dispersed zinc ferrite ($ZnFe_2O_4$) nanoparticles. They studied the effects of types and amounts of the added salt on the properties of the products were investigated by XRD, SEM, and BET nitrogen adsorption techniques. The introduction of salt into reaction system facilitates the formation of well-dispersed spinel zinc ferrite and increases specific surface area of the resultants from 7.14 to 88.34 m^2 g^{-1} when KCl/M ratio is 2/3.

Nanocrystalline $Mg_{(1−x)}ZnxFe_2O_4$ (x=0.0, 0.25, 0.5, 0.75, and 1) ferrites were synthesized recently by a solution combustion method using a mixture of fuels, i.e., sugar and urea. The effect of zinc content was studied, and an increase in saturation magnetization, magnetic moment, and remnant magnetization was found with an increase in the zinc content up to x=0.5 [104].

Fig. 2 Stages of combustion reaction of magnesium ferrite liquid solution of magnesium nitrate, iron nitrate, and urea at room temperature. From (**b**) to (**d**), the hot blanked temperature is c.a. 300 °C. **b** Water started to boil and evaporating from the solution, which substantially increased solution viscosity. **c** The emerging intensive flame indicates that combustion reaction has been started. The reaction lasts less than 30 min. **d** The combustion reaction is entirely completed, and the final product is powder of nanoparticles of $Mg_{0.2}Fe_{0.8}O$. Reprinted from [102] an open access
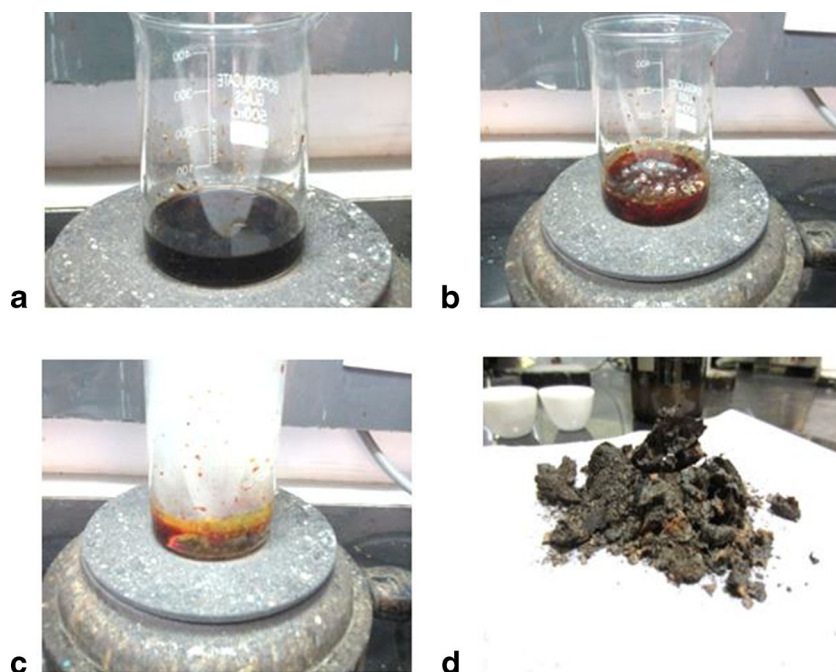
Co-precipitation

Co-precipitation is a facile and convenient way to synthesize MNPs (metal oxides and ferrites) from aqueous salt solutions by the addition of a base under inert atmosphere at room temperature or at elevated temperature. The size, shape, and composition of the magnetic nanoparticles very much depend on the type of salts used (e.g., chlorides, sulfates, nitrates), the Fe^{2+}/Fe^{3+} ratio, the reaction temperature, the pH value, and the ionic strength of the media [105]. Iron oxide nanoparticles (either Fe_3O_4 or γ-Fe_2O_3) and ferrites are usually prepared in an aqueous medium which chemical reaction of formation may be written as Eq. 1

$$M^{2+} + 2Fe^{3+} + 8OH \rightarrow MFe_2O_4 + 4H_2O \qquad (1)$$

where M can be Fe^{2+}, Mn^{2+}, Co^{2+}, Cu^{2+}, Mg^{2+}, Zn^{2+}, and Ni^{2+}. Complete precipitation should be expected at pH levels between 8 and 14, with a stoichiometric ratio of 2:1 (Fe^{3+}/M^{2+}) in a nonoxidizing oxygen environment [106].

Nickel-doped cobalt ferrite magnetic nanoparticles (19–32 nm) were synthesized in a homogeneous aqueous solution in the presence of surfactant at different calcination temperatures by co-precipitation route [107]. Shinde et al. [108] prepared nanocrystalline spinel ferrites with general formula $Ni_{1-x}Zn_xFe_2O_4$ (x¼0–1.0) by an oxalate co-precipitation method. The saturation magnetization increases with increasing concentration of zinc up to x¼0.4 and then decreases with increasing zinc concentration.

Iwasaki et al. [109] developed a simple novel process for preparing Fe_3O_4 nanoparticles by a co-precipitation route without using any additives (e.g., surfactant and oxidizing and reducing agents). In this method, a cooled ball mill was used as a synthesis reaction field in order to inhibit progress of both the synthesis reaction and the particle growth by heat energy. The Fe_3O_4 nanoparticles were formed by ball milling of the starting suspension consisting of ferrous hydroxide and goethite colloids, and the crystallization was simultaneously progressed without heating.

Magnetic Fe_3O_4 nanopowder was synthesized by ultrasonic-assisted chemical co-precipitation utilizing high purity iron separated from iron ore tailings by acidic leaching method. Surfactant influence on particles shape and size was investigated. Fe_3O_4 nanoparticles coated with $C_{12}H_{25}OSO_3Na$ exhibit better dispersion and uniform size [110].

Sharma et al. [50] adopted the simple co-precipitation technique for the synthesis of single-phase ZnO:Co^{2+} nanoparticles of mean size 2–8 nm and studied their magnetic and optical properties.

Microemulsion technique

Microemulsion has also been widely used to synthesize uniformly sized MNPs [111–116]. This is a thermodynamically stable isotropic dispersion of two immiscible liquids, where the microdomain of either or both liquids is stabilized by an interfacial film of surfactant molecules [117]. The surfactant molecule lowers the interfacial tension between water and oil resulting in the formation of a transparent solution. Depending on relative concentrations, surfactant molecules self-assemble into a variety of structures in the solvent mixture, such as micelles, bilayers, or vesicles. Most commonly used structures in nanoparticles synthesis are micelles, either as reverse (water-in-oil) or normal (oil-in-water) form. In both cases, the dispersed phase consists of monodisperse droplets in the size range of 2–100 nm [118]. This dispersed phase provides a confined environment for the synthesis and formation of nanoparticles [119].

Lee et al. [120] carried out large-scale synthesis of uniform and highly crystalline magnetite nanoparticles from the reaction salts in microemulsion nanoreactors. They have controlled the particle size from 2 to 10 nm by varying the relative concentrations of the iron salts, surfactant, and solvent.

Nanoparticles of barium ferrite ($BaFe_{12}O_{19}$) were synthesized by Pillai et al. [121] using microemulsion processing. In this method, the aqueous cores (typically 5–25 nm in size) of water-cetyltrimethylammonium bromide-n-butanol-octane microemulsions were used as constrained microreactors for the co-precipitation of precursor carbonates (typically 5–15 nm in size). The carbonates thus formed were separated, dried, and calcined to form nanoparticles of barium ferrite. The schematic representation of reaction in microemulsions is given in Fig. 3.

Nickel zinc ferrite nanoparticles ($Ni_{0.20}$ $Zn_{0.44}$ $Fe_{2.36}$ O_4) have been produced at room temperature, without calcination, using a reverse micelle process by Morrison et al. [122]. $CoFe_2O_4$ nanoparticles have been synthesized by microemulsion method through the formation of normal micelles using sodium dodecyl sulfate (NaDS) as a surfactant by Liu et al. [123].

Other methods of synthesis

There are certain other methods for the synthesis of magnetic nanoparticles. These include thermal decomposition [43], sonochemical [124], electrochemical [125], bacterial synthesis [126, 127], polyol method [128, 129], and so on. Zhao et al. [130] carried out water-soluble superparamagnetic Fe_3O_4 nanoparticles with an average diameter of 9.5 ± 1.7 nm which were synthesized by thermal decomposition of Fe(acac)$_3$ in methoxy poly(ethylene glycol) (MPEG). MPEG was used as solvent, reducing agent, and modifying agent in
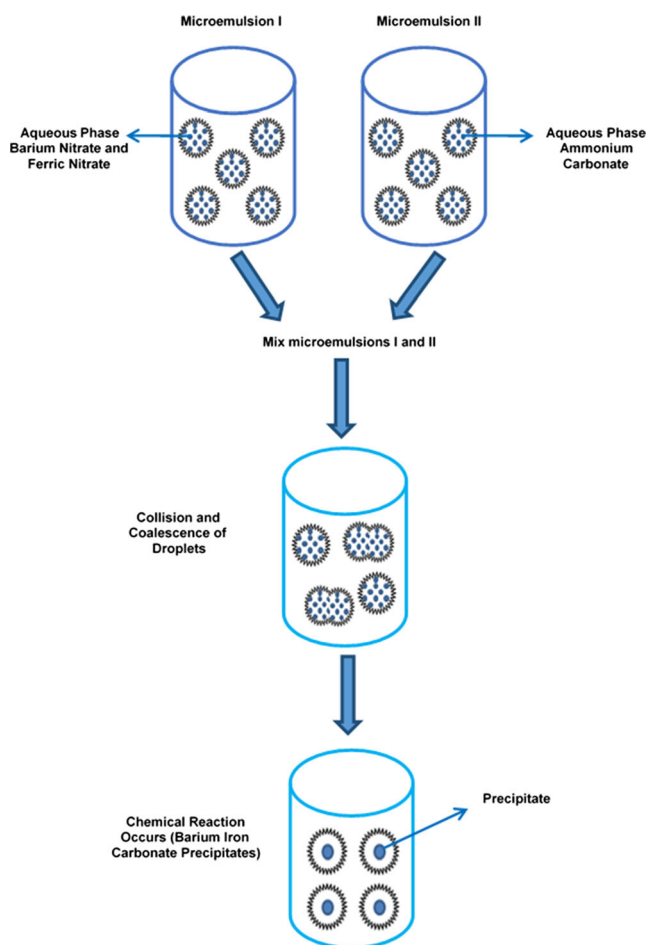
Fig. 3 Schematic representation of the reaction in microemulsions. Reprinted with modification from [121], Copyright 1993, with permission from Elsevier

this reaction. A tentative mechanism of the formation of Fe_3O_4 nanoparticles is shown in Fig. 4.

Mazar′ıo et al. [131] obtained uniform size $CoFe_2O_4$ ferrite nanoparticles in one step using an electrochemical technique. Recently, superparamagnetic $ZnFe_2O_4$ nanoparticles with size range of 28–38 nm were synthesized by polyol process based on use of varying chain length glycols (diethylene and polyethylene glycol) as solvent [132].

Properties of magnetic nanoparticles

Magnetic nanoparticles exhibit magnetic properties based on their magnetic susceptibility (χ) which is defined as the ratio
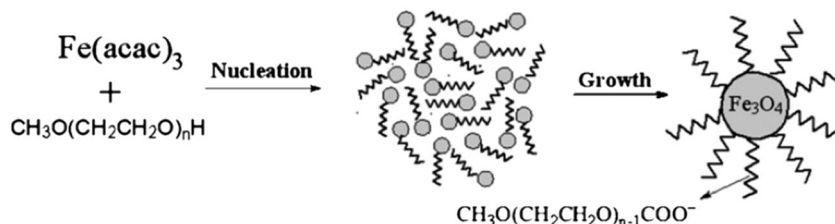
of induced magnetization (M) to the applied magnetic field (H). Ferromagnetic materials possess aligned atomic magnetic moments of equal magnitude, and coupling interactions between the electrons of the material result in ordered magnetic states (e.g., Fe, Ni, and Co). Furthermore, susceptibilities of these materials depend on their atomic structures, temperature, and external field H. As the size decreases to the order of tens of nanometers, ferri- or ferromagnetic materials become a single domain and therefore maintain one large magnetic moment. However, at sufficiently high temperatures, thermal energy is sufficient to cause free rotation of the particle (brown relaxation) and relaxation of the magnetic spins (Neel relaxation) resulting in the loss of net magnetization [133, 134]. In this state, materials exhibit paramagnetic behavior. Lack of remnant magnetization after removal of external fields enables the particles to maintain their colloidal stability and avoids aggregation making it feasible for their use in biomedical applications. The coupling interactions in single magnetic domains result in much higher magnetic susceptibilities than paramagnetic materials. Ferrimagnetism is a property exhibited by materials whose atoms or ions tend to assume an ordered but nonparallel arrangement in zero applied field below Neel temperature usually within a magnetic domain (e.g., Fe_3O_4 and Fe_3S_4). Figure 5 shows the magnetization behavior of ferromagnetic and superparamagnetic nanoparticles (NPs) under an external magnetic field [133]. A substantial net magnetization results from the antiparallel alignment of neighboring nonequivalent sub-lattices. The macroscopic behavior is similar to ferromagnetism. Above the Neel temperature, the substance becomes paramagnetic [135, 136].

In addition with magnetic properties, magnetic nanoparticles also show structural, optical, electrical, and gas sensing properties. A number of studies on these properties of magnetic nanoparticles have been reported in the literature [89, 130, 137–141].

Applications of magnetic nanoparticles

Magnetic nanoparticles have found a plethora of applications in a wide variety of scientific fields [142]. Some of the general applications of magnetic nanoparticles are presented in Fig. 6.

Iron oxides are widely used as low-cost adsorbents for removal of heavy metal ions [84, 143–146], organic dyes [147–149], drug delivery [150], cell labelling and sorting [151], magnetic resonance imaging, sensing [152, 153], as

Fig. 4 The illustration of the magnetite nanoparticles prepared by thermal decomposition in MPEG. Reprinted from [130], Copyright 2007, with permission from Elsevier
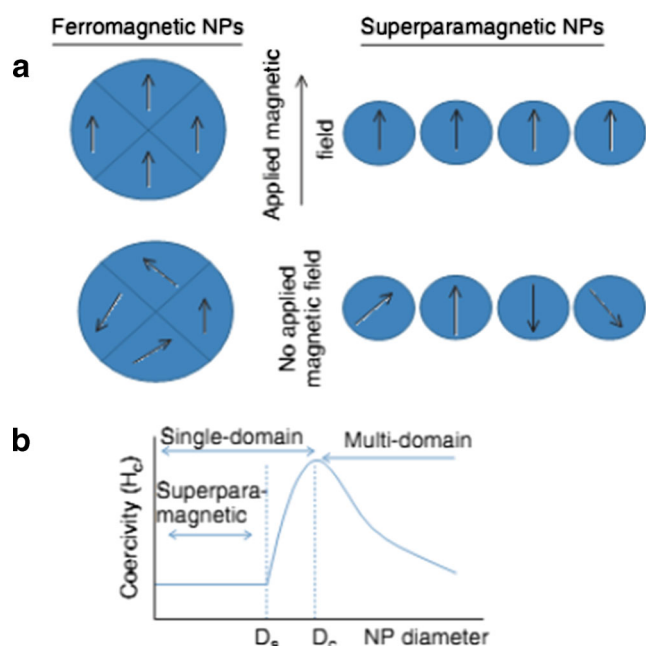
Fig. 5 Magnetization behavior of ferromagnetic and superparamagnetic NPs under an external magnetic field. **a** Under an external magnetic field, domains of a ferromagnetic NP align with the applied field. The magnetic moment of single domain superparamagnetic NP aligns with the applied field. In the absence of an external field, ferromagnetic NPs will maintain a net magnetization, whereas superparamagnetic NPs will exhibit no net magnetization due to rapid reversal of the magnetic moment. **b** Relationship between NP size and the magnetic domain structures. D_s and D_c are the "superparamagnetism" and "critical" size thresholds. Reprinted from [133], Copyright 2012, with permission from Springer

well as therapeutic applications such as AC magnetic field-assisted cancer therapy, i.e., hyperthermia [154–156]. Hematite and magnetite have also been used as catalysts to catalyze a number of important reactions, such as synthesis of NH_3,
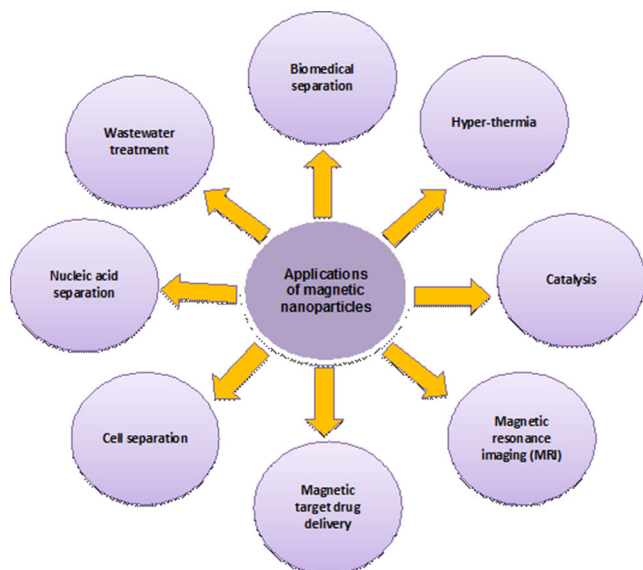
Fig. 6 General applications of magnetic nanoparticles (MNPs). Reprinted with modification from [142], Copyright 2011, with permission from Wiley

desulfurization of natural gas and high-temperature water-gas shift reaction, Fisher-Tropsch synthesis for hydrocarbons, dehydrogenation of ethylbenzene to styrene, oxidation of alcohols, and large-scale synthesis of butadiene [44, 157, 158]. Spinel ferrites also cover a wide range of applications, such as ferrofluids [159], microwave absorber [160], magnetic drug delivery [161], magnetic resonance imaging (MRI) contrast [162], permanent magnets, hard disk recording media, sensors [163], and catalysis [164]. Dilute magnetic semiconductors are very promising for spintronic device applications [165–167]. Polymer magnets have uses in computer hardware, for example as disk drives and in medical devices such as pacemakers and cochlear implants where the organic material is more likely to be biocompatible than its metallic counterpart.

Magnetic nanocomposites

Types of magnetic nanocomposites

There are mainly three types of magnetic nanocomposites: (i) core–shell inorganic nanocomposites, (ii) self-assembled nanocomposites, and (iii) organic–inorganic nanocomposites.

1. Core–shell inorganic nanocomposites

    The combination of two nanoscale entities into a single hybrid particle has recently attracted much attention due to the numerous possibilities of application [168]. Hybrid nanoparticles may provide a platform with dual imaging capabilities for medical diagnosis, dual action combining magnetic imaging and therapy, and multiplexing in sensors. By this approach, the respective properties of the components may be combined and optimized independently. In addition, cooperatively enhanced performances due to collective interactions between the constituents have been achieved.

    Silica (SiO_2) has been employed as a very common and promising material for coating and encapsulating magnetic NPs. There are several reasons for choosing SiO_2 as coating or matrix material in magnetic composite materials. First of all, it reveals silanol surface groups which may easily be derivatized with a variety of functional groups, providing a convenient platform for further functionalization. SiO_2-based magnetic composites have been studied in view of various biomedical and environmental applications due to their biocompatibility, stability against degradation, and a hydrophilic character. In addition, there are procedures to manufacture mesoporous SiO_2 materials with well-defined pores and large surface areas which have a great impact on catalytic and separation purposes. Magnetic SiO_2 composite materials have

been prepared by several procedures, including aerosol routes (e.g., aerosol pyrolysis, spray-drying), microemulsion polymerization, and sol–gel processes [169].

2. Self-assembled colloidal nanocomposites

The self-assembly of small building blocks (e.g., atoms, molecules, and nanoparticles) into ordered macroscopic superstructures has been an important issue in various areas of chemistry, biology, and material science. Self-assembly of NPs into two-dimensional and three-dimensional superlattices with a high degree of translational order has attracted a lot of attention [170]. These super crystals did not only reveal a translational order but further an orientational order with a crystallographic alignment. The assembly of NPs of different materials into defined colloidal crystals or quasi crystals provides a general path to a large variety of composite materials (metamaterials) with new collective properties arising from the interaction of the different nanocrystals in the assembly [170].

3. Organic–inorganic nanocomposites

This is the classical type of a nanocomposite, where the isolated NPs are finely dispersed in a polymer. In reality, agglomerated NPs are dispersed in a polymer matrix. Functional nanocomposites with improved physical properties allow various applications (e.g., in biomedical, microoptics, electronics, energy conversion, or storage). In most of the cases, the change of the aspired feature correlates with the filler load. The resulting composite flow behavior limits mostly huge solid loadings and therefore property adjustment due to restrictions in shaping or molding. Shear rate and temperature-dependent as well as oscillatory rheological investigations are therefore necessary for a detailed description of the composite flow properties prior to shape forming [171]. In case of nanosized fillers, the specific surface area and the resulting huge polymer-filler interfacial layer dominates the rheological behavior. In this chapter, we mainly concentrated on the synthesis and properties of organic–inorganic hybrid nanocomposites.

Synthesis of magnetic polymer nanocomposites

The synthesis of organic–inorganic hybrid nanocomposites composed of magnetic materials and polymers has gained increasing attention due to their potential applications [105, 172]. Advances in polymer science have demonstrated the ability to prepare a vast array of materials exhibiting controllable mechanical, thermal, and electroactive properties. As part of this renewed interest in nanocomposites, researchers began seeking new strategies to engineer materials that combine the desirable properties of nanoparticles and polymers for

the formation of nanocomposites. The research revealed a number of key challenges in producing nanocomposites with the desired behavior. The greatest hindrance to the large-scale production and commercialization of nanocomposites is the absence of cost-effective methods for controlling the dispersion of the NPs in polymeric hosts. The nanoscale particles typically aggregate, which cancels out any benefits associated with the dimension [173]. The particles must be integrated in a way leading to isolated, well-dispersed primary nanoparticles inside the matrix. There is a need for establishing processing techniques that are effective on the nanoscale yet are applicable to macroscopic processing. Synthetic strategies for nanocomposites with a high homogeneity are, therefore, really a challenge. There have been several attempts for the synthesis of nanocomposites that can be classified under two major categories: ex situ and in situ processes as will be discussed in the following sections. In this chapter, the in situ methods are discussed in detail and examples are given. However, it is impossible to completely describe this field due to the vast number of published papers on the synthesis of magnetic nanocomposites. Therefore, this chapter will give a general overview of the techniques and strategies used for the preparation of magnetic nanocomposites. Selected examples that represent different routes and systems will be reported. More detailed descriptions on specific themes will be referred from related references.

Ex situ method

The traditional and simplest method of preparing organic–inorganic nanocomposites is direct mixing of the fillers into the polymer. The mixing can generally be done by melt blend in gand solution blending. The ex situ method is a popular one because it does not set a limitation on the nature of nanoparticles and host polymers to be used.

Direct mixing of particles with the polymer melt in technical polymer processes like extrusion is the classical method for the preparation of composite materials from thermoplastic polymers. It is widely used for the compounding of clay materials in polyolefins. Melt compounding is currently explored to a wide range of materials such as oxides and carbon nanotubes. Strength of melt compounding is the large quantity of material that can be produced by extrusion, since most polymer blends are commercially produced in this way. Melt blending is a cost-effective widely used method in industry that involves the intimate mixing of the filler into the polymer matrix. There were only very few works reported based on the synthesis of magnetic nanocomposites by melt blending. Wilson et al. [174] synthesized polymer nanocomposites of poly(methyl methacrylate) (PMMA) doped with varying concentrations of Fe NPs (~20 nm). These nanocomposites were processed by melt blending using the C.W. BrabenderPlasticorder® at a temperature of 210 °C, slightly

above the PMMA's melting temperature of 200 °C. The mixture was melt blended for 5 min; this produced the master batch. Nanocomposites (0.5 and 1.0 %) were made by melt blending a portion of the master batch with neat PMMA. The same group also synthesized Fe/polystyrene (PS) nanocomposites by melt blending [175]. They have developed a controlled and reproducible way of synthesizing polymer magnetic nanocomposites with the potential for scaling up the production to meet the larger quantity requirements for electromagnetic interface (EMI) shield and other possible magnetic and radiofrequency applications. Kong et al. [176] prepared magnetic nanoparticle-natural rubber nanocomposite by melt blending and studied the temperature-dependent magnetic behavior of the nanocomposite. Shannigrahi et al. [177] prepared PMMA/ferrite powder $[(Ni_xR_{1-x})_{0.5}Zn_{0.5}Fe_2O_4$ (R=Mn, Co, Cu; x=0, 0.5)] nanocomposite films by melt blending. A dry mixture of ferrite powders and polymers was placed inside the melt extruder to prepare rod-shaped composites, which were further hot pressed to produce composite films. Among these, cobalt-doped ferrites and their composites showed the best EMI shielding effectiveness value and have potential for practical EMI shielding applications. Rajput et al. [178] synthesized flexible magnetic nanocomposites composed of cobalt ferrite-polybenzoxine-linear low-density polyethylene by melt blending. These nanocomposites showed structural flexibility and magnetic properties. Chung et al. [179] prepared flexibly cross-linked shape memory polyurethane/Fe_3O_4 magnetic composites by melt mixing. The composites displayed excellent mechanical and shape memory properties. Vunain et al. [180] prepared polymer nanocomposites, constituted of ethylene-vinyl acetate, polycaprolactone, and Fe_3O_4 with different weight percentage, by melt blending using a Thermo Scientific HaakeRheomex OS extruder equipped with roller rotors. The nanocomposites were used as effective adsorbent materials for the removal of As(III) ions and some metal ions from aqueous solutions. Although polymer processing conditions were optimized to achieve good uniform dispersion of the NPs in the polymer matrix, surface characterization indicated the clustering of nanoparticles. The agglomeration is attributed to the particle interaction mediated by steric force in the polymer matrix. Overall, the reasonable dispersion and control over magnetic properties achieved in the above reports are promising for electromagnetic applications of these materials. However, blending polymers and nanoparticles to afford homogeneous and well-dispersed NPs in the polymer pose significant challenges.

Solution blending or solvent casting process consists of physical entrapment of the metal or metal oxide NPs in the polymer network proceeds through casting and solvent evaporation, chemical polymerization, or co-precipitation. Such encapsulation of NPs also helps in the stabilization of NPs by preventing them to agglomerate. In this process, the magnetic NPs are preformed and mixed with a solvent and a polymer solution. After the solvent is cast either in a form of membrane or coating on another surface, low-grade heat is utilized to evaporate the solvent, leaving behind the nanocomposite material [181–183]. For the solvent casting, the polymer synthesis step can be separated from the particle generation and the nanocomposite processing. Therefore, more complex polymer architectures are accessible. In addition to the synthetic polymers, biopolymers such as chitosan, alginate, etc. have been widely used for ex situ synthesis of the nanocomposite [184–186]. Table 1 provides few examples of the

Table 1 Ex situ synthesis of magnetic polymer nanocomposites

Nanomaterials	Polymer used	Synthesis	Reference
Fe/Fe-oxide	PMMA	The NPs were dispersed by sonication into a solution of PMMA powder in acetone. The solution was then spin cast until it hardened into a thin film	[181]
Fe_3O_4	[Styrene-b-ethylene/butylene-b-styrene)]	Fe_3O_4/polymer nanocomposites were prepared using a solution-casting method. Oleic acid-modified Fe_3O_4 and block copolymer were dissolved in THF, and then a film was cast over a period of 1 day	[182]
γ-Fe_2O_3/Fe_3O_4 mixed phases	(Poly(ethylmetha crylate-co-methylacrylate)	NPs were dispersed in the copolymer solubilized in chloropharm. After being sonicated, the resulting solution was drop casted on a glass substrate	[183]
Fe_3O_4	Chitosan	NPs were dispersed in the chitosan solution by stirring at room temperature after which it was sonicated. Finally, a highly viscous solution of chitosan with uniformly dispersed Fe_3O_4 NPs was obtained. The nanocomposite film was prepared by the dispersion of solution into ITO	[184]
Fe(III)/Ni(II) hydroxides	Alginate	To the homogeneous hydroxide suspension, sodium alginate powder was slowly added and the mixture was stirred until the total polymer dissolution was achieved. A peristaltic pump was used to dispense the suspension in a stirred reservoir containing $CaCl_2$ solution used for gel formation	[185]
Fe_3O_4	Polydimethylsiloxane	Oleic acid-functionalized Fe_3O_4 NPs were dispersed in prepolymer. Then, the polymer cross-linker was added and mixed. The final nanocomposite solution was casted into a mold and thermally cured	[186]

THF tetrahydrofuran, *ITO* indium tin oxide

magnetic nanocomposites synthesized by ex situ method [181–186].

In situ method

Many ex situ processes generally suffer extremely from the high agglomeration tendency of nanoparticles, as it is rather difficult to destroy the nanoparticle agglomerates even using high external shear forces. In situ nanoparticle formation and in situ polymerization methods have been developed to overcome this problem.

In the in situ particle formation method, inorganic precursors and polymers were dissolved in a suitable solvent. The inorganic NPs were in situ formed via chemical reactions in the polymer matrix. A simple method for the synthesis of nanocomposite is through the hydrolysis or reduction of metal salts in the presence of a polymer. This method makes it possible to manipulate the polymer/inorganic filler interfacial interactions at various molecular and nanometer scales, resulting in homogeneous nanocomposites and thus overcoming the problem of NP agglomeration.

The sol–gel process is a wet chemical technique widely used in the fields of materials science and ceramic engineering. The term sol–gel is associated with two reaction steps, sol and gel. A sol is a colloidal suspension of solid particles in a liquid phase and gel the interconnected network formed between the phases. The sol–gel processing includes two approaches: hydrolysis of the metal alkoxides and then polycondensation of the hydrolyzed intermediates. Hydrolysis involves the cleavage of the organic chain bonding to metal and the subsequent replacement with –OH groups through nucleophilic addition. The protonated species leave the hydrolyzed metal as an alcohol. Condensation is based on oxygen, metal, and oxygen bond formation (–O–M–O–). Materials prepared by sol–gel processing have a higher uniformity, higher purity, and low sintering temperature than that of conventional solid state reaction. The biggest problem of sol–gel is that the gel process would lead to a considerable contraction of the internal stress which could result in contraction of brittle materials, due to the evaporation of solvents, small molecules, and water. Moreover, this method requires polymers in sol–gel system be dissolved in the condensate. In addition, the precursors are expensive and sometimes toxic, preventing the further improvement and application.

A promising method for the in situ growth of particles in the presence of a polymer is the thermal decomposition of metal precursors forming metal or metal oxide NPs within the polymeric matrix which then acts as a stabilizer. Polymeric stabilizers can be designed with tunable composition via copolymerization or post-functionalization techniques. Such polymeric surfactants must include a few general features, such as functional groups to anchor the particle surface (e.g., carboxylic acid, amine, or amide groups) located either along the polymeric backbone and/or at the end. In the early 1990, Ziolo et al. [187] elaborated a one-step chemical method to synthesize fine dispersed γ-Fe$_2$O$_3$ NPs in a cross-linked PS resin. They used a synthetic ion exchange resin and aqueous solutions of Fe(II) or Fe(III) chloride, respectively, to exchange the ions. Cao et al. [188] synthesized Fe$_3$O$_4$/PMMA composite particles by a one-pot hydrothermal method. A wide range of polymers has been used to synthesize and colloidally stabilize magnetic NPs via polymeric coatings [189, 190]. For example, polystyrene was used as a surfactant in the thermolysis of dicobalt octacarbonyl to afford uniform ferromagnetic nanoparticles [191]. Magnetic cobalt nanocrystals were prepared via a reverse micellar synthesis followed by a hydrothermal treatment. An aqueous solution of cobalt chloride and poly(sodium 4-styrenesulfonate) was mixed first with an organic mixture containing cetyltrimethylammonium bromide to form reverse micelles, followed by reducing cobalt ions with sodium borohydride. The resultant nanoparticles were then undergone a hydrothermal treatment to generate well-dispersed cobalt nanocrystals [192].

The procedure of in situ polymerization involves dispersing the inorganic NPs directly in the monomer or monomer solution and subsequent polymerization of the monomer dispersion with standard polymerization techniques. Polymer coating on the surface of magnetic NPs enhances the compatibility with organic moiety, reduces the leaching of the NPs, and protects them from oxidation. Synthesis of polymer encapsulated magnetic NPs was pioneered by Ugelstad et al. [193] in 1993. The methodology was based on direct precipitation of iron salts inside the pores of porous PS particles. As a result, PS particles with magnetic NPs encapsulated inside the pores were produced. Up to date, various synthetic strategies for the preparation of polymer/magnetic particle composites have been reported including suspension crosslinking, dispersion polymerization, and emulsion/inverse emulsion polymerization.

Suspension cross-linking approach has been reported as a simple and an efficient method for encapsulation of magnetic NPs in the polymer matrix. Denkbas et al. [194] prepared magnetic particle-encapsulated chitosan composite by suspension cross-linking approach. Stable magnetic emulsion droplets were first prepared by rigorous stirring the heterogeneous mixture containing aqueous-based magnetic nanoparticles, chitosan, emulsifier, and diethyl either. Subsequently, the chitosan in the emulsion droplets was cross-linked by the addition of glutaraldehyde. Other researchers also prepared chitosan/magnetic NP composites by the same technique [195, 196]. Besides chitosan, albumin and poly(vinyl alcohol) were employed for the encapsulation of magnetic NPs [197, 198]. However, the resulting composite particles were microsized with broad size distribution.

Dispersion polymerization is a simple and fast (single step) technique used to prepare monodisperse polymer particles of a range of sizes (1–20 μm) with very high yields [199]. Dispersion polymerization is a type of precipitation polymerization, and the reaction is performed in the presence of a suitable polymeric stabilizer that is soluble in the reaction medium. The selected continuous medium is a solvent for the monomer to be polymerized and a nonsolvent for the resultant polymer. A steric stabilizer is used to produce a colloidally stable dispersion, without which the polymerization would generate macroscopic polymer particles of uncontrolled size. Horak and Benedyk [200] prepared poly(glycidylmethacrylate)/Fe$_2$O$_3$ composite by dispersion polymerization in aqueous alcoholic media using poly-(N-vinylpyrrolidone) and azobisisobutyronitrile as a steric stabilizer and initiator, respectively. Fan et al. [201] synthesized composite microspheres which consists of poly(maleic anhydride-co-methacrylic acid) [P(MAH-MAA)] copolymer as a shell and Fe$_3$O$_4$ modified with bi-surfactants, sodium dodecyl benzene sulfonate, and oleic acid as a core with improved magnetic properties (Fig. 7).

One of the promising techniques for the encapsulation of magnetic NPs into polymer matrix is using microemulsion polymerization. Daniel et al. [202] prepared magnetic polymer particles by first mixing organic-based magnetic NPs with hydrophobic vinyl monomer and emulsifier. The polymerization resulted in a magnetic NP as a core and polymer as a shell. A similar technique was used by Charmot et al. [203] to prepare core–shell composite with the addition of a vinyl cross-linker. Dresco et al. [113] reported a one-step synthesis of magnetic composites by inverse emulsion technique. Inverse microemulsion droplets were first prepared by mixing water-soluble monomer (methacrylic acid, hydroxyethyl methacrylate), cross-linker, surfactant, aqueous-based magnetic NPs in toluene. Copolymerization of these emulsion droplets gave stable composites. Wormuth et al. [204] also used this approach for the encapsulation of magnetic NPs in the diblock copolymer [poly(ethylene oxide-co-methacrylic acid)] matrix. However, the composite particles produced by emulsion or inverse emulsion methods had a very broad size distribution. In addition, it was very difficult to remove the residual surfactants adsorbed on the resulting particle surface.

Miniemulsion is a thermodynamically stable homogeneous liquid in which stable oil drops with sizes ranging from 50 to 500 nm are dispersed in an aqueous continuous phase. The oil drops are confined by surfactant micelles and a highly water-insoluble compound (i.e., hydrophobe). Pamirez et al. [205] prepared core–shell Fe$_3$O$_4$/PS composite by miniemulsion polymerization using hexadecane and sodium dodecyl sulfate (SDS) as hydrophobe and emulsifier, respectively (Fig. 8). The composite particles had narrow size distribution. Luo et al. [206] also prepared PS/Fe$_3$O$_4$ composite particles via miniemulsion polymerization in the presence of potassium persulfate as an initiator, SDS as a surfactant, and hexadecane or sorbitan monolaurate as a co-stabilizer. Xu et al. [207] reported the magnetic core–shell composites via inverse miniemulsion polymerization of acrylamide and a cross-linker in the presence of poly(methacrylic acid)-coated magnetic NPs. However, this technique requires a large quantity of surfactant and hydrophobe.

Grafting techniques take advantage of monomers being directly adsorbed to the particle surface. In "grafting to" techniques, polymer chains modified with anchoring groups are used to bind to the particle surface. Polymers can be grafted to the inorganic particle by reacting these anchoring groups with functional sites at the particle surface.

"Grafting from" approach includes monomer adsorption onto the particles followed by a subsequent polymerization catalyzed by an initiator. This technique uses initiators that have been initially anchored to the particle surface, followed by the polymerization from the surface. A scheme describing this technique is presented in Fig. 9 [208]. Table 2 shows various examples of the synthesis of nanocomposites using these two methods [209–216].

Surface-initiated atom transfer radical polymerization (ATRP) is one of the most robust and versatile approaches for the synthesis of polymer films. ATRP is a transition metal-catalyzed, controlled ("living") radical polymerization mechanism, offering the advantage of good control on the molecular weight and polydispersity. Vestal and Zhang [217] were the first to report the surface-initiated ATRP of styrene from a MnFe$_2$O$_4$ NPs system. A microemulsion method was used to synthesize SDS-stabilized MnFe$_2$O$_4$ NPs, which were exchanged with 3-chloropropionic acid to prepare the colloidal

Fig. 7 Schematic representation for preparation of core–shell micro/nanostructured P(MAH-MAA)/Fe$_3$O$_4$ composite microspheres. Reprinted from [201], Copyright 2009, with permission from Springer
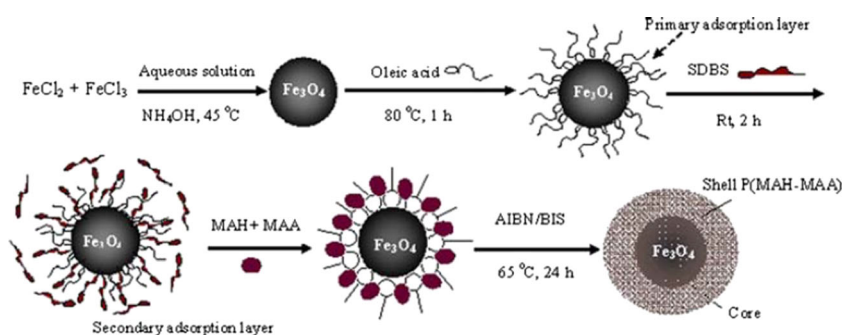
Fig. 8 Formation of core–shell Fe$_3$O$_4$/PS composite by miniemulsion polymerization. Reprinted from [205], Copyright 2003, with permission from Wiley-VCH Verlag GmbH & Co. KGaA
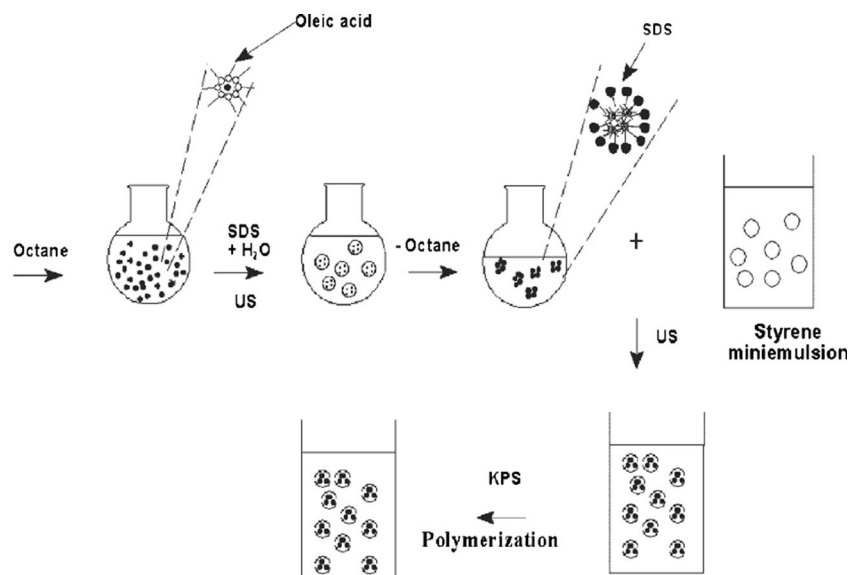
initiator. Heterogeneous styrene ATRP using a copper(I) chloride (CuCl)/2,2′-dipyridyl catalyst yielded the hybrid particle with the PS shell and the MnFe$_2$O$_4$ core. The preparation of core–shell PS grafted γ-Fe$_2$O$_3$ NPs was also reported by Wang et al. [218] using a combination of ligand exchange and surface-initiated ATRP. Using a similar strategy, Duan et al. [219] conducted the surface-initiated ATRP of DMAEMA from γ-Fe$_2$O$_3$ NPs. The use of silane coupling agents to covalently anchor ATRP initiating sites onto the iron NPs was demonstrated to prepare PMMA-coated core–shell colloids [213, 220]. Thermo-responsive magnetic core–shell NPs were prepared by Gelbrich et al. [221] via surface-initiated ATRP of 2-methoxyethyl methacrylate from a colloidal Fe$_3$O$_4$ initiator. Via surface-initiated ATRP, iron oxide NPs were also coated by a copolymer shell of oligo(ethylene glycol) methylether methacrylate (OEGMA) and MEMA with adjustable thermo-responsive behavior in water Fig. 10 [222]. Surface-initiated nitroxide-mediated polymerizations have also been investigated to prepare PS or poly(3-vinylpyridine)/Fe$_3$O$_4$ core–shell composites [223]. A recent report by Wang et al. [224] cited the use of reversible addition-fragmentation

chain transfer technique to incorporate PS or poly(acrylic acid) chains onto Fe$_3$O$_4$ NPs via surface-initiated polymerization.

Other techniques

Recently, there are many reports on the use of special methods for preparing polymer/inorganic nanocomposites such as plasma polymerization, layer by layer method, and seed precipitation method.

The plasma polymerization technique is a room temperature and environmentally benign process. The main principle of the plasma polymerization technique is that the ionized and excited molecules and radicals created by the electrical field bombard react with the surface of the substrate. A versatile approach for the gas-phase synthesis of hybrid core–shell NPs is the application of microwaves for plasma generation. This approach was developed by Vollath et al. [225, 226]. The basic element of this approach is a reaction tube made of quartz glass crossing a microwave cavity. At this intersection, plasma is ignited. Volatile and water-free precursors (e.g.,

Fig. 9 Schematic description of grafting to and grafting from approaches for the synthesis of PINCs. Reprinted from [208], Copyright 2003, with permission from Elsevier
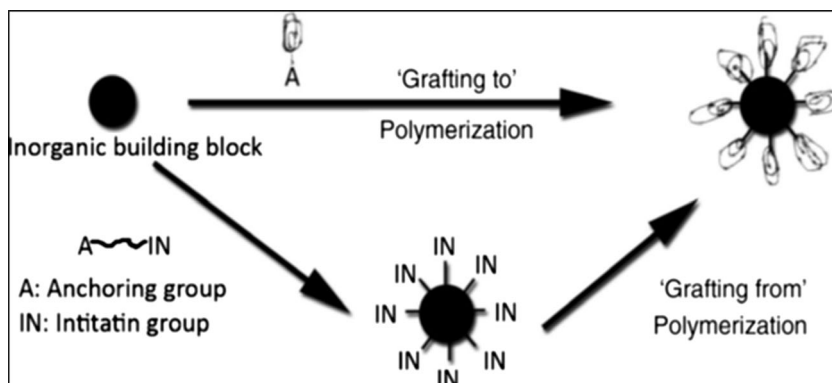
Table 2 Synthesis of magnetic polymer nanocomposite by "grafting to" and "grafting from" approaches

| Method	Core	Shell	Anchor	Reference
Grafting to	Fe_3O_4	PEG	APS	[209]
	γ-Fe_2O_3	PCL	IPTS	[210]
	γ-Fe_2O_3	PVI	MPS	[211]
	$NiFe_2O_4$	PVA	–	[212]
Grafting from	γ-Fe_2O_3	PMMA	CDS	[213]
	γ-Fe_2O_3	PCL	AAPM	[214]
	γ-Fe_2O_3	PEG	MPS	[215]
	Fe_3O_4	PDMAEMA	BIB	[216]

PCL poly(ε-caprolactone), *AAPM* N-(2-aminoethyl)-3-aminopropyltrimethoxysilane, *CDS* (chloromethyl)phenylethyldimethylchlorosilane, *PEG* polyethylene glycol, *MPS* methacryloxypropyltrimethoxysilane, *PDMAEMA* poly(2-(dimethylamino)ethylmethacrylate), *BIB* N-bromoisobutyrate acid, *APS* aminopropyltrimethoxysilane, *IPTS* 3-isocyanatopropyltriethoxysilane, *PVI* poly(1-vinylimidazole), *PVA* poly(vinyl alcohol)

chlorides, carbonyls, metal alkoxides, or metal-alkyls) are evaporated outside the reaction tube and mixed with an inert carrier gas. The components are introduced as gases into the system just in front of the plasma zone. Here, the chemical reaction in the gas phase and the nucleation and growth of NPs occurs. By using consecutive reaction zones, core–shell NPs and multilayer NPs can be produced in consecutive synthesis steps. The inorganic cores are formed by homogeneous nucleation, and the organic shell of hybrid nanoparticles condenses via heterogeneous nucleation and polymerizes outside of the plasma zone on the cores synthesized in the plasma. A

Fig. 10 Synthesis of thermo-responsive magnetic iron oxide-polymer brush particles via surface-initiated ATRP, thermo-flocculation of the particles in water. Reprinted from [222], Copyright 2010, with permission from Elsevier

similar approach was used by others [227, 228] to synthesize core–shell nanocomposites.

Layer by layer deposition of NPs and polymers can also be utilized for the formation of highly homogeneous thin composite films. Caruso et al. [229] developed the controlled synthesis of novel magnetic core–shell nanocomposites that consist of alternating layers of magnetic nanoparticles and polyelectrolytes as a shell and PS as a core. Core–shell polymer template was first prepared by deposition of three layers of poly(allylamine hydrochloride) (PAH) onto PS particle surface. Magnetic NPs (10 to 15 nm) were then adsorbed onto the polymer template through electrostatic complexation. Alternating deposition of PAH and magnetic NPs resulted in the formation of multilayer shells on the template surface. However, this method is time consuming due to sequential polyelectrolyte and magnetic NP depositions and purification cycles. In addition, the leaching problem of magnetic NPs from the inner shell layer and the stability of the shell layers to pH and electrolyte changes may still be of concern.

Seed precipitation polymerization has been developed for the preparation of core–shell composite microparticles. Sauzedde et al. [230, 231] prepared the hydrophilic temperature-sensitive magnetic latexes. Seed magnetic NPs were prepared by adsorption of negatively charged Fe_3O_4 NPs onto positively charged colloidal template [PS/poly(N-isopropylacrylamide)]. The encapsulation was performed via copolymerization of N-isopropylacrylamide and N,N-methylene bisacrylamide, itaconic acid in the presence of the seed particles. The resulting particles had a diameter of around 500–800 nm but with narrow size distribution. Zaitsev et al. [232] also prepared composite particles via seed precipitation polymerization of methacrylic acid and hydroxyethyl methacrylate in the presence of tris(hydroxyl methyl)aminomethane hydroxide-coated magnetic NPs. The resulting particles had a diameter of around 150 nm but with broad size distribution. Gu et al. [233] prepared vinyl terminated magnetic NP-encapsulated PS composite. However, in the seed precipitation polymerization, encapsulation process is quite difficult to be controlled [234].
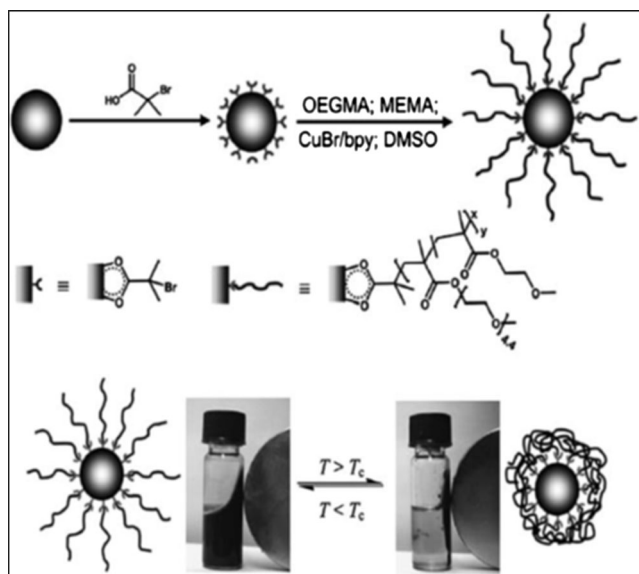
Applications of magnetic polymer nanocomposites

Environmental applications

Removal of heavy metals

Industrial wastes from various industries such as paints and pigments, mining and extraction, glass production, plating, and battery manufacturing plants release various metals in to water bodies. These metals are not biodegradable and their presence in water causes bioaccumulation in living organisms,

leading to diseases such as cancer, kidney failure, metabolic acidosis, oral ulcer, and renal failure. Many of them though not toxic, but their presence in nonpermissible doses can create health problems. Heavy metal ions as Cd^{2+}, Cr^{3+}, Cu^{2+}, Pb^{2+}, Zn^{2+}, As^{3+}, or Hg^{2+}, originating from natural geological or industrial sources, cause hazardous water pollution [235].

Effective removal of undesirable metals from water systems is very important but still challenging task for environmental researchers. A number of traditional as well as modern technologies, such as chemical precipitation [236], ion exchange processes [237], membrane filtration [238], reverse osmosis [239], electrochemical treatment [240], coagulation [241], extraction [242], irradiation [243], biological methods and adsorption [244], have been employed for the treatment of heavy metal-contaminated wastewater. However, adsorption [245] is regarded as the most promising technique because of its high efficiency, simplicity, and cost-effectiveness. Several types of materials, such as activated carbons, clay minerals, biopolymers, chelating materials, and zeolites, have been exploited to adsorb metal ions from aqueous solutions. However, challenge has been there for removal of adsorbent-containing metal ions. If core of adsorbent is magnetic, then separation is facilitated. The removal of metal ions using magnetic adsorbents has recently grabbed attention for wastewater treatment. Figure 11 represents how magnetic separation facilitates the removal of heavy metals using magnetic nanocomposites.

Lead ions commonly exist in industrial and agricultural wastewater. Long-term drinking water containing high level of lead ions cause serious disorders, such as nausea, convulsions, coma, renal failure, cancer, and detrimental effects on metabolism and intelligence [246].

Applications of different nanosized magnetic particles for the removal of metal ions from water were studied. The studied materials include maghemite, magnetite (Fe_3O_4), diatomite supported/unsupported magnetite nanoparticles, and surface-modified jecobosite nanoparticles [247–250]. The adsorbing power of inorganic magnetic nanomaterials is poor, but their incorporation into organic polymers and biomolecules can enhance the adsorption capacity. Moreover, the stronger ionic interaction between metal ions and composite surface occurs through pore diffusion that primarily enhances sorption–desorption dynamics. The separation is further facilitated due to magnetic core. The magnetic nanocomposite based on organic polymers and biopolymers thus offers a better alternative for conventional metal removal techniques because of ease of separation and administration.

Fan et al. [251] synthesized magnetic nanocomposites of chitosan and graphene oxide by a facile and fast process. The stable and environment-friendly composite was used to adsorb Pb^{2+} ions from aqueous medium with adsorption capacity of 76.94 mg/g. In a similar study, chitosan-coated magnetite nanoparticles were successfully used to remove Pb^{2+} ions [252]. A 53.6 % removal efficiency was reported. The magnetization value of 70.1 m^3 Kg^{-1} was achieved for Fe_3O_4 nanoparticles without chitosan, while composites were characterized by a decrease in magnetization (66.4 and 45.1 m^3 Kg^{-1}) with an increase in chitosan content. Fe_3O_4/ cyclodextrin polymer nanocomposites were used for selective heavy metal removal from industrial wastewater. Carboxymethyl-β-cyclodextrin polymer-modified Fe_3O_4 nanoparticles was synthesized for selective removal of Pb^{2+}, Cd^{2+}, and Ni^{2+} ions from water. Grafting of polymer on MNPs enhanced the adsorption capacity because of the complexing abilities of the multiple hydroxyl and carboxyl groups in polymer backbone with metal ions [253]. Fe_3O_4-coated glycine-doped polypyrrole magnetic nanocomposites were prepared via coating of suspended Fe_3O_4 nanoparticles with glycine-doped polypyrrole. Results showed that magnetic nanocomposites are effective adsorbents for the removal of Cr(VI) from wastewater [254]. Hybrid magnetic nanocomposites were prepared via anchoring the Fe_3O_4@polypyrrole nanospheres with hierarchical porous structure on graphene nanosheets. These magnetic nanocomposites exhibit excellent adsorption capability for Cr(VI) removal due to the synergic effect between graphene and Fe_3O_4@polypyrrole. Resulting magnetic nanocomposites could be a good candidate for

Fig. 11 Pictorial representation of magnetic separation of adsorbed metal ions from aqueous medium using magnetic nanocomposite
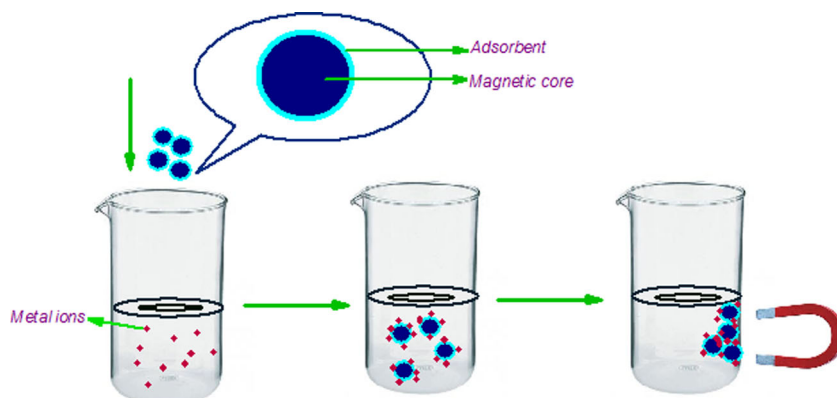
efficient Cr(VI) removal from the wastewater [255]. Magnetic Fe_3O_4 nanoparticle surface modification was carried out with an amino-terminated silane coupling agent. A copolymer of methyl methacrylate and maleic anhydride was transformed into magnetic nanocomposite by chemical immobilization of surface-modified Fe_3O_4 with the anhydride groups of copolymer chains. Sorption of metal ions to magnetic nanocomposites agreed well with the Langmuir adsorption model with the maximum adsorption capacity of 90.09, 90.91, 109.89, and 111.11 mg g^{-1} for Co^{2+}, Cr^{3+}, Zn^{2+}, and Cd^{2+}, respectively [256]. A novel amino-functionalized magnetic cellulose composite was prepared by coating magnetic silica nanoparticles with cellulose followed by grafting of glycidyl methacrylate and ring-opening reaction of epoxy groups with ethylenediamine to yield amino groups. Synthesized nanocomposite was tested for its ability to remove Cr(VI) from an aqueous solution, and results demonstrated that Cr(VI) adsorption was highly pH dependent. The adsorption isotherms of the adsorbent fit the Langmuir model with the maximum adsorption capacity of 171.5 mg/g at 25 °C. It was reported that resulting composite material can be a promising adsorbent for Cr(VI) removal, with the advantages of high adsorption capacity, rapid adsorption rate, and convenient recovery under magnetic field [257]. Magnetic hydrogel beads consisting of carboxylated cellulose nanofibrils, amine-functionalized magnetite nanoparticles, and poly(vinyl alcohol)-blended chitosan were prepared by an instantaneous gelation method and used as adsorbents for Pb(II). Carboxylate groups of carboxylated cellulose nanofibrils play an important role in Pb(II) adsorption, and magnetic hydrogels exhibit higher adsorption capacity with the value of 171.0 mg/g [258]. Magnetic carbonaceous polysaccharide nanocomposites were synthesized through hydrothermal process as potential adsorbents for water treatment. Adsorption behavior of nanocomposites for metallic ion contaminants was observed by batch adsorption procedure. Superparamagnetic property of magnetic polymer nanocomposites makes them recyclable for adsorption application [259].

Radioactive pollution is among the other hazards. Radioactive cesium contamination is of serious social and environmental concerns. Magnetic hexacyanoferrate (II) polymeric nanocomposites were prepared for separation of cesium from low-level radioactive wastes. Polyacrylonitrile was used as a binding polymer. It was found that maximum separation was attained within 2 h of contact, and cesium exchange was independent from pH value [260].

Removal of toxic dyes and effluents

The rapid industrialization and urbanization has resulted in the discharge of dyes and other colored toxic effluents into water bodies. The presence of these dyes in water is highly visible and undesirable. They are produced to be resistant to weather, light, water, and detergent. Moreover, the dyes are either not biodegradable under aerobic conditions or they degrade slowly via conventional biological processes, producing vividly colored treated effluents. These dyes are harmful to aquatic plants and animals, and some of the organic dyes have a mutagenic and carcinogenic action on human beings [261]. The most trusted method to remove dyes from aqueous solutions is adsorption as it is easy and cost-effective [262–264]. Secondly, advanced oxidation process using nano photocatalysts is currently the most exploited technique for organic pollutant removal by generation of hydroxyl free radicals [265–269]. However, it is very difficult in complete elimination of nanoparticles from the water after the purification, which results in leaching of the nanoparticles into the environment. Such problems can be solved by the use of magnetic recoverable photocatalysts and adsorbents. Magnetic separation has advantages such as its easy phase separation with aqueous solutions and its capability of treating large amount of wastewater within a short time. Photocatalysts or bio-adsorbents supported on magnetic carriers, magnetic photocatalysts supported by organic materials, and magnetic polymer nanocomposites are among the materials involved for dye removal. Biopolymers and organic adsorbent-based magnetic nanocomposites have been successfully utilized previously for the decontamination of the organic dyes from wastewater [270–274]. Pourjavadi et al. [275] studied the removal of crystal violet dye using magnetic nanocomposite $Fe_3O_4@SiO_2$ starch-*graft*-poly(acrylic acid) hydrogels. The magnetic hydrogels were easily separated because of high magnetic sensitivity. A 85 % or higher degradation of dye was achieved.

Among biopolymers, chitosan has been extensively used for removal of dyes via adsorption because of high functionality. Methylene blue was removed using magnetic chitosan/graphene oxide nanocomposite by adsorption [276]. The adsorption fitted Langmuir adsorption isotherm, and a maximum adsorption capacity of 180.83 mg/g was reported. In a similar study, Zhu et al. [277] exploited magnetic chitosan/poly(vinyl alcohol) hydrogel beads (saturation magnetization (21.96 emu g^{-1})) for adsorption of Congo red from aqueous solution. The equilibrium adsorption capacity was found to be 470.1 mg/g which was higher than chitosan/carbon nanotube nanocomposite [278]. Ferrite-based nanocomposites are also used extensively for dye removal. Polymer [polyvinyl pyrollidine (PVP), polyethylene glycol (PEG), polyvinyl alcohol (PVA)]-functionalized $Zn_{0.5}Co_{0.5}Al_{0.5}Fe_{1.46}La_{0.04}O_4$ magnetic nanocomposites were found to be more effective in dye decolorization than the ferrite itself [279]. The dye absorption efficiency for ferrite was 76 and 84–90 % for composites. Fe_3O_4 nanoparticles as the core of magnetic nanomaterials have also been proven to be biocompatible with low toxicity. Therefore, many adsorbents based on the magnetic nanoparticles were synthesized through surface

modification with various organic compounds, such as chitosan, cationic biopolymer, anionic biopolymer, ionic liquid, and surfactants. Effective adsorption of Rhodamine B dyes was observed using a magnetic nanocomposite of Fe_3O_4 and gum-ghatti an anionic natural polysaccharide. Authors reported a maximum adsorption capacity of 471.69 mg g^{-1} and catalyst regeneration of five times [280]. Various other magnetically separable nanocomposites were used for dye removal either by adsorption or photocatalysis such as MWCN–starch–iron oxide composites [281].

Oil removal

Wastewater containing oils in the form of fats, lubricants, petroleum products, and cutting oils is one of the environmental concerns nowadays. Due to an increase in oil exploration over the world in accordance with rise in demands for oil has made removal of oils an issue of technological importance. Oil spills in the water bodies pose a threat to aquatic life [282]. Though oil spills are normal accidents, they are on rise in world as per various reports from international agencies. The oils present in emulsion form are more difficult to tackle with because of high stability in aqueous medium [283].

The use of floating barriers is the most commonly used technique to control the spread of oil, whereas sorbents are used to remove final traces of oil or in areas that cannot be reached by skimmers. The techniques as adsorption [284, 285], coagulation [286], coalescence [287], and membrane filtration [288] have been used by researchers worldwide to separate oils from water. Natural organic adsorbents like cellulose, fibers, peat moss, etc. have been used for adsorption of

oil. In recent times, advanced functional nanomaterials comprising of organic and inorganic counterparts have gained importance [289, 290]. The conventional techniques are expensive and separation is not quite easy. Magnetic materials having affinity for oil are an excellent alternative. However, magnetic materials as Fe_3O_4, ferrites, etc. have no affinity for oils. Hence, they are to be functionalized or modified by species which can facilitate the adsorption of oil.

The Fe_3O_4 nanoparticles coated with polystyrene (Fe_3O_4@PS) were synthesized [291] as a material with higher affinity for oil and water repellent action. The composite adsorbed lubricating oil three times its weight. The digital photographs of the adsorption of oil and magnetic recovery by Fe_3O_4@PS are shown in Fig. 12. Due to hydrophobic organic counterpart in organic–inorganic magnetic nanocomposites, oil gets easily adsorbed and magnetically removed or recovered due to magnetic core inside.

Oily wastewater purification was done using polymethyl acrylate (PMA)-functionalized magnetite nanoparticles, and recovery was performed by superconducting magnetic separation. The composite showed a high ferromagnetism and outstanding ability to absorb oil [292]. Fe_3O_4, as a significant member of magnetic nanoparticle class, has found applications in catalysis, magnetic storage, magnetic separation, etc. Polymers exhibit unique properties due to their large surface areas, high chemical stability, hydrophobicity, and high functionality. Thus, composites based on Fe_3O_4 and polymers have been most exploited for oil removal applications. In similar works, magnetic polystyrene nanocomposites [293] and Fe_2O_3 in alkyd resin [294] were used for recovery of oil from oil-contaminated water.

Fig. 12 Digital photograph images of removal of lubricating oil from water surface by Fe_3O_4@PS nanocomposites under magnetic field. The lubricating oil was labelled by Sudan I dye for clarity. Reprinted from [291], Copyright 2013, with permission from Elsevier
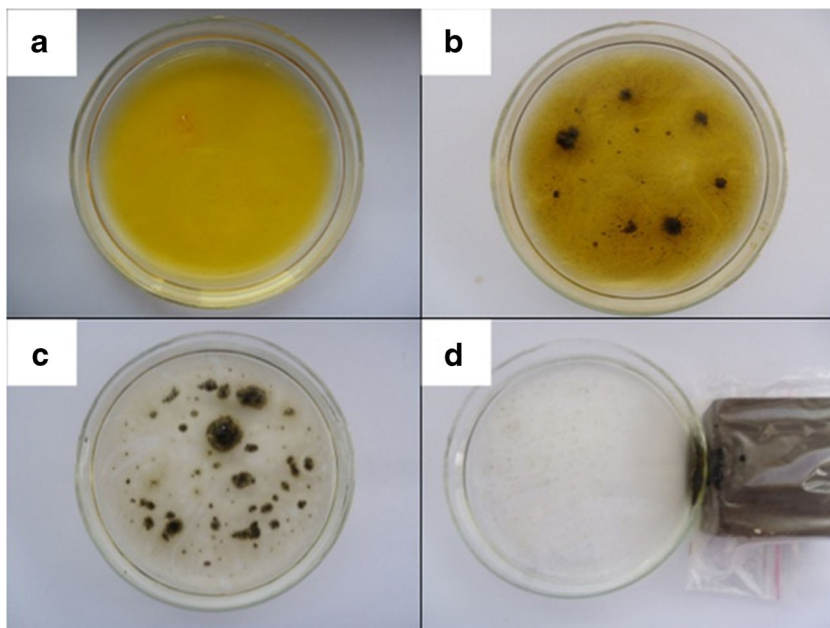
Biomedical applications

Targeted drug delivery

Magnetic materials with nanoscale dimensions have been explored in the field of targeted drug delivery because they have a strong potential to improve diagnosis and treatment. MNPs as drug carriers [295–297] are unique as they can be directed and localized under the influence of magnetic field. Among magnetic nanoparticles, SPIONs have proved to be most successful as diagnostic and therapeutic agents. Addition of bioactive and organic molecules to the SPION surface can make them more target specific [298–300] and provide tailored treatment by producing contrast agents that specifically differentiate between affected and healthy tissues.

Polymer nanocomposites exhibiting superparamagnetic behavior have been recognized as a promising tool to achieve targeted drug delivery using external magnetic field for treating complex diseases. Release of a therapeutic agent at specific site and rate is achieved by means of composite nanoparticles, consisting of the carrier, the bound, or encapsulated bioactive payload and surface modifiers. Chen et al. [301] synthesized a biocompatible magnetic nanocomposite of silica, iron oxide, and polymethacrylic acid and used anticancer drug doxorubicin as model drug. A drug loading efficiency of 105 ± 8 μg/mg of drug carrier was reported. Kappa carrageenan-g-poly(acrylic acid)/SPION nanocomposite was exploited as targeted drug delivery system for release of the drug deferasirox. The composite was also found effective as an in vitro antibacterial agent [302].

Superparamagnetism is very useful in drug delivery because SPIONs can be transported by electrical field effects to the desired site, and they remained at target site even after removal of magnetic field. Polymer nanocomposite of SPION and polyvinyl alcohol modified with polymethyl methacrylate were used for delivery of popular drug ciprofloxacin. It was reported that release was accelerated in the presence of magnetic field but with lower amounts of PVA and higher amounts of PMMA [303].

In general, magnetic targeted drug delivery involves the attachment of a cytotoxic drug to a biocompatible carrier (magnetic nanoparticles functionalized by biocompatible molecules). It is followed by intravenous injection of these carriers in the form of a colloidal suspension, application of a magnetic field to direct the carrier to the target site, and release of the drug. Though the process seems to be simple, there are many complications involved. Various parameters as field strength, depth of the target tissue, and blood flow play an important role for successful delivery.

Cancer diagnosis and therapy

Magnetically guided drug targeting using magnetic nanoparticles and nanocomposites is a promising approach for cancer chemotherapy and diagnosis. The use of biocompatible magnetic nanocomposites as drug or gene delivery systems can contribute to the effectiveness of cancer therapy in many ways. Magnetic nanocomposites are used in diagnosis by MRI and sensing, treatment by drug delivery and gene targeting, and prevention by vaccination and heat delivery by hyperthermia. This has been pictorially represented in Fig 13. Among the broad spectrum of nanobiomaterials under investigation for cancer comprehensive treatment, magnetic nanocomposite materials have gained significant attention due

Fig. 13 Pictorial illustration of exploited and potential use of magnetic nanocomposites in cancer diagnostics and treatment
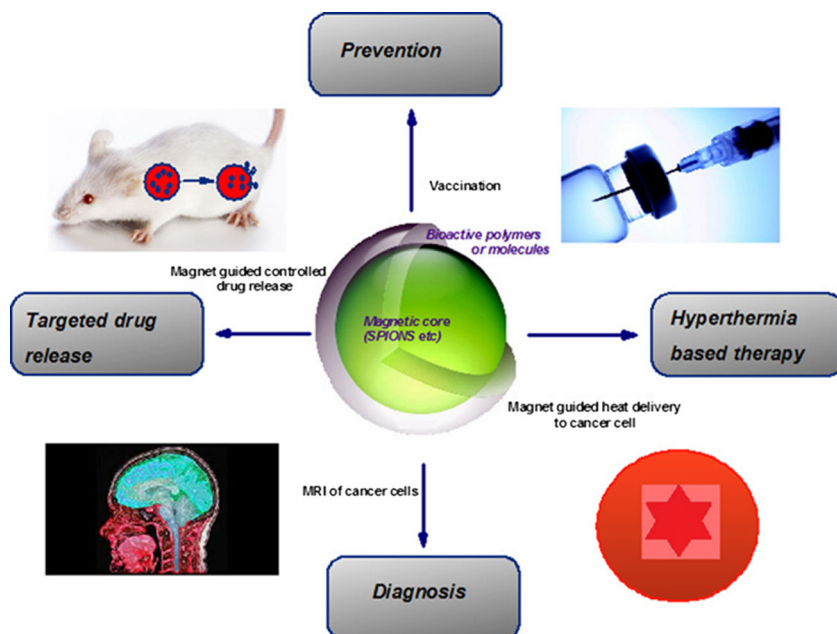
to their unique features which are not present in other materials. In particular, superparamagnetic particles are the preferred ones. SPIONs find applications in cancer treatment [304–307], tissue repair [308], and magnetic cell sorting [309, 310]. One of the most intriguing applications of SPIONs is as an MRI contrast agent for cancer diagnosis. The early detection of tumor/cancer markers in blood or tissue provides easy disease diagnosis, disease recurrence, and treatment for long-term survival of cancer patients.

Magnetic materials are advantageous over conventional nanoparticles in cancer targeting because their behavior allows monitoring and quantitative determination of their biodistribution by MRI. Sufficient penetration of therapeutic agent into cancer cell is achieved, and targeting of tumors with magnetically guided nanoparticles is site specific. SPIONs coated with dextran [311, 312], PVA [313], and PEG [314] have been successfully used in magnetic resonance imaging of cancer cells.

In 2009, Hoare and colleagues achieved therapeutic release in thermo-sensitive poly(N-isopropylacrylamide)-based nanogels and magnetic nanoparticles [315]. A magnetic thermo-sensitive hydrogel based on magnetite chitosan and β-glycerophosphate was developed by Zhang and co-workers [316] for delivery of Bacillus Calmette–Guerin (BCG) for treatment of bladder cancer. Magnetic injectable hydrogels significantly prolonged the intravesical BCG residence time under an applied magnetic field. BCG delivered by the gel system induced a stronger Th1 immune response and revealed higher antitumor efficacy in comparison to traditional BCG therapy. Magnetic drug targeting has also been reported in various research works using Fe_3O_4/chitosan [317], silica-coated magnetic polystyrene containing CdTe/CdS and

Fe_3O_4 nanoparticles [318], and magnetite/chitosan-L-glutamic acid [319].

Magnetic resonance imaging and bioimaging

MRI technique is based on the counterbalance between the small magnetic moment on a proton and the exceedingly large number of protons present in biological tissue, which leads to noticeable change in the presence of magnetic fields [320, 321]. Iron oxide nanoparticles are the most commonly used contrast agents because they are superparamagnetic, biocompatible, and possess high saturation magnetization and dispersibility.

However, MNPs suffer a serious disadvantage of aggregation or agglomeration. Therefore, modification of these nanoparticles is necessary using inorganic [322, 323] or organic materials [324, 325]. Zhu et al. [326] developed pH-sensitive nanocomposite composed of SPION bonded to antitumor drug doxorubicin and poly(ethylene glycol) combining MRI diagnosis and drug release together. The T_1 and T_2 values show fourfold higher increase as compared to free magnetite nanoparticles showing enhanced MR contrast both in in vivo and in vitro studies. Recently, fullerenes and graphene have been exploited in MR imaging due to their extraordinary properties. In a recent work, polyethylene glycol-modified fullerene/iron oxide nanocomposites were synthesized by Shi and co-workers [327] and used for in vivo MRI on female C57 mice having B16-F10 tumors and reported the composite as a negative (T_2) contrast agent. Similarly, polyethyleneimine-mediated synthesis of folic acid-targeted Fe_3O_4 nanoparticles was reported for in vivo MRI of tumors. Folic acid-targeted Fe_3O_4 has a high T_2 relaxivity of

Fig. 14 Fluorescence stability of MPN–QDs–SiO$_2$ antibodies on the surface of MDA-MB-435S cancer cells. The cells were continuously excited by the upright fluorescent microscope with the light wavelength of 488 nm. The cells were excited for **a** about 30 s, **b** 15 min, **c** 30 min, and **d** 60 min, respectively. *Bar*, 10 μm. Reprinted from [318], Copyright 2011, with permission from Springer
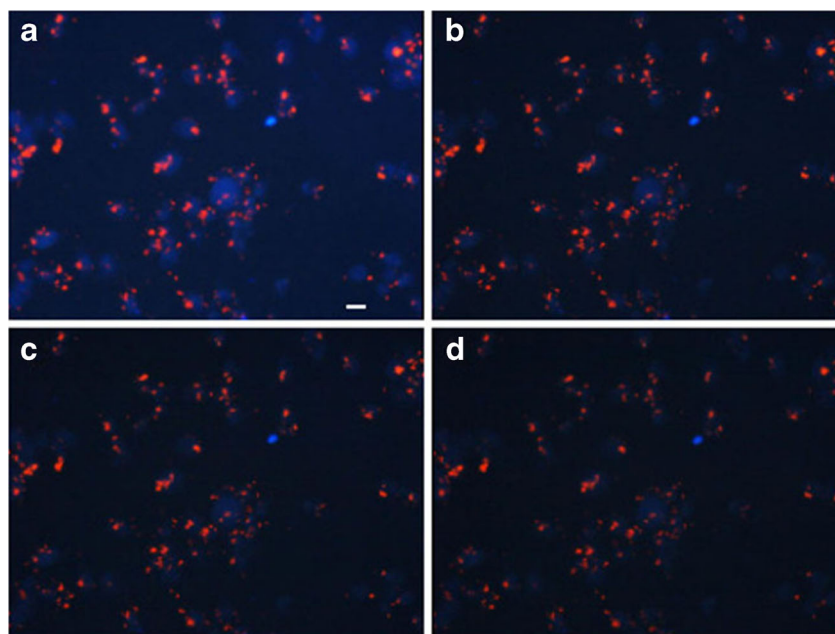
99.64 mM^{-1} s^{-1} and acts as a nanoprobe for MRI of cancer cells [328].

Theragnostics is referred to the fusion of therapeutic and diagnostic techniques. Magnetic nanoparticles can be designed to encapsulate a wide variety of chemotherapeutic and diagnostic agents for dual purpose of imaging of tumor cells and therapeutic treatment. Such study was made by Xu et al. [329] by developing polymer encapsulated up conversion nanoparticles/iron oxide nanocomposites having excellent photoluminescence and magnetic properties and used for both targeted drug delivery and image-guided therapy. Similar observations were reported by Ling and co-workers [330].

Biocompatible PEG-modified, phospholipid-coated iron oxide nanoparticles were functionalized by a fluorescent dye and peptide and then used for the confocal imaging of primary human dermal fibroblast cells and kidney-derived cells. These nanocomposites possessed a great potential for tissue imaging [331]. Silica-coated magnetic polystyrene nanospheres (MPN) containing CdTe/CdS QDs and Fe_3O_4 nanoparticles were prepared, and antibodies were conjugated onto these magneto-fluorescent nanocomposites (MPN–QDs–SiO$_2$) for cancer cell targeting and imaging. The fluorescence of MPN–QDs–SiO$_2$ antibodies-labelled cancer cells (MDA-MB-435S) was recorded. Figure 14 shows that nanoparticle fluorescence on the cells did not change for 30 min on excitation of labelled cells by fluorescent light (488 nm) for 15, 30, and 60 min, which was useful for long-term observations [318].

Conclusions

Magnetic nanoparticles and nanocomposites are interesting materials because of their potential applications in the fields of biomedicine, information technology, magnetic resonance imaging, catalysis, telecommunication, and environmental remediation. Grafting of or coating with organic species, including surfactants or polymers, can stabilize the naked magnetic nanoparticles and avoid their aggregation. Hybrid magnetic nanocomposites are interesting materials because of the dual character of organic and inorganic counterparts, and a variety of methods are being used for their synthesis. Magnetic polymer nanocomposites found utilization in environmental and biomedical application such as removal of heavy metals, removal of toxic effluents, oil removal, targeted drug delivery, magnetic resonance imaging, cancer diagnosis, and therapy.

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